

JAPANESE

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CLAIMS DETAILED DESCRIPTION TECHNICAL
FIELD PRIOR ART TECHNICAL PROBLEM
MEANS EXAMPLE DESCRIPTION OF DRAWINGS
DRAWINGS

[Translation done.]

* NOTICES *

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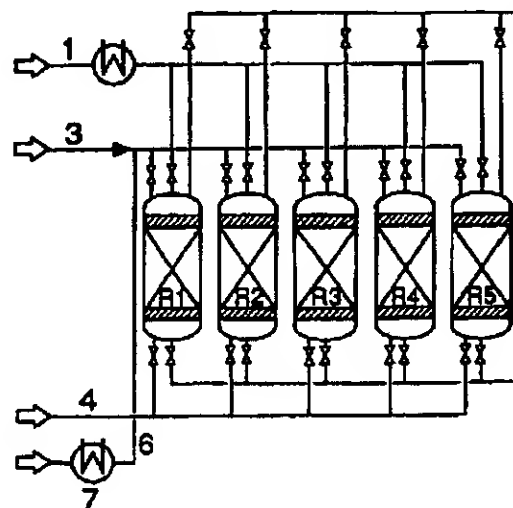

CLAIMS

[Claim(s)]

[Claim 1] In the method of manufacturing chlorine from the gas which contains oxygen and a hydrogen chloride by reacting at the temperature raised in the reactor containing a dee contest catalyst at the a 1st process When the gas containing oxygen and a hydrogen chloride is introduced into a reactor, the temperature in a reactor is raised and only 10-90 degrees C reacts a dee contest catalyst and a hydrogen chloride The chlorine compound fixed to water and the catalyst is formed, water is discharged from a reactor, and after the 1st process is completed, a reactor is washed by inert gas. succeedingly at the b 2nd process The manufacture method of chlorine which the chlorine compound formed beforehand is made to react, and supplies the oxidizer of the gas which enables isolation of chlorine to a reactor, discharges the chlorine which separated from a reactor, and is characterized by washing a reactor by inert gas succeedingly.

[Translation done.]

Drawing selection

[Representative drawing] 

[Translation done.]

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention relates to the method of having been improved which manufactures chlorine from the gas containing oxygen and a hydrogen chloride by reacting at the temperature raised in the reactor containing a dee contest catalyst (Deacon catalyst).

[0002]

[Description of the Prior Art] A hydrogen chloride is formed at many reactions as a by-product which is not desirable, and, for this reason, research of the latus range and the proposal of a method already exist about reprocessing of this by-product. As an example the reaction in which a hydrogen chloride is formed as a by-product is indicated to be, manufacture of a vinyl chloride or an isocyanate and erasure of a contaminant are mentioned.

[0003] The one possible method of reworking a hydrogen chloride is a reaction which forms chlorine, and can be reused by the manufacturing process by making chlorine into a raw material or an intermediate product.

[0004] The method of various form is used for this deformation.

[0005] In hydrochloric-acid electrolysis, chlorine is manufactured by electrolysis of an aquosity hydrogen chloride. This method needs the output of most amount for a complicated electrolysis cell and electrolysis. Therefore, it is complicated in method engineering and cost starts.

[0006] The method of other common knowledge is based on a Deacon process. A hydrogen chloride is made to react with air under existence of a catalyst as follows by this method.

[0007] $4\text{HCl} + \text{O}_2 \rightleftharpoons 2\text{H}_2\text{O} + 2\text{Cl}_2$ -- reference -- for example, -- Weissermel K., Arpe H.-J., and Industrielle Organische Chemie -- the 3rd edition 1988. 233 - 234 pages, VCH Verlagsgesellschaft Weinheim, Allen J.A. Clark A.J. "Oxychlorination Catalysts", and Rev.Pure and Appl.Chem.21 (1971) 145-166 A page and Furusaki S. "Catalytic Oxidation of Hydrogen Chloride in a Fluid Bed It is indicated by Reactor", AIChE Journal, and 19(5) (1973) 1009-1016 page in the latus range.

[0008] In D1 (U.S. Pat. No. 2204733 specification), a Deacon process is enforced in a fixed bed reactor (the 38th 2-page line to the 64th line, and drawing 3). However, the high heat of a reaction produces a remarkable local temperature rise in a fixed catalyst bed, and since a reaction balance moves in the direction of a starting material, this decreases the invert ratio of a reaction. To conquer this difficulty by the inversion of a flow inside the fixed bed was tried by D1. However, movement of the concentration through the catalyst which has remarkable volatility at the temperature produced all over a floor (some component existing as a melt corrosive in the place which has very high temperature), therefore can hardly be controlled by the vapor pressure inside the fixed bed produces the catalyst used. This heterogeneity produces reduction in an invert ratio further. Furthermore, a fixed catalyst bed may be closed by the catalyst which deposited.

[0009] The fluidized bed process which tried to prevent formation of the big foam which is not desirable with the punched board is indicated by D2 (the Europe patent public presentation No. 331465 specification) (the three pages of the 56th line from the 55th 2-page line, drawing 1). It does not pass over the invert ratio attained using this method to at most 83%, but the temperature in a reactor is about 360-400 degrees C.

[0010] However, by corrosive [of the high dynamical load produced in a fluid bed reactor, and a starting material / high], the reactor required for this method is technically complicated, and requires cost. Furthermore, an unreacted hydrogen chloride and the worn matter must be discharged from the fluid bed, therefore it must process, having to spend many costs, and the catalyst loss by discharging must be filled up.

[0011] The fluidized bed process which the aforementioned fault produces also in D3 (the Europe patent public notice No. 474763 specification) is indicated.

[0012] The fluidized bed process of many processes is indicated by D4 (U.S. Pat. No. 2577808 specification) (the 5th column of the 18th line from the 3rd column of the 65th line, drawing). A catalyst bed passes through a chlorination band behind a preheating or a cooling-zone region, and forms the catalyst compound which the hydrogen chloride reacted with the catalyst here and was chlorinated. The catalyst compound chlorinated in the continuing oxidizing-zone region reacts with oxygen further, and

separates chlorine. A catalyst bed is recycled in the 1st band by pneumatic pressure.

[0013] The wear which is not desirable cannot be avoided in this fluidized bed process, and corrosion cannot be completely prevented under existence of a corrosive starting material by the high mechanical load of a reactor. Furthermore, in order for an invert ratio to decrease by post-mixture operation and to improve yield, it must operate at temperature which is different in each band, and this complicates this method further. Furthermore, risk of a hydrogen chloride, chlorine, or water being conveyed in other reaction-zone regions exists. Furthermore, it is a fault that the processing which chlorine content process exhaust gas produced by this method, therefore was complicated for this reason is required.

[0014] The method of two processes of manufacturing chlorine from HCl is indicated by D5 (the Europe patent public notice No. 500728 specification). It dechlorinates at the temperature of at least 300 degrees C by chlorinating at the remarkable low temperature of 25-250 degrees C (claim 1). (dechlorination) This needs continuous heating or cooling of a lot of solids (from the 54th line to the 9th column, the 16th line to the 19th line, and the 58th line). This demand restricts possibility of manufacturing a corrosion-resistance reactor further.

[0015]

[Problem(s) to be Solved by the Invention] The technical problem of this invention was finding out the method the aforementioned fault's being canceled and chlorine's being manufactured still more easily in method engineering.

[0016]

[Means for Solving the Problem] The aforementioned technical problem is the temperature raised in the reactor containing a dee contest catalyst, it is solved by the method of manufacturing chlorine from the gas which contains oxygen and a hydrogen chloride by reacting at 300-450 degrees C advantageously, and this method is the a 1st process. When the gas containing oxygen and a hydrogen chloride is introduced into a reactor, the temperature in a reactor is raised and only 10-90 degrees C reacts a dee contest catalyst and a hydrogen chloride The chlorine compound fixed to water and the catalyst is formed, water is discharged from a reactor, and after the 1st process is completed, a reactor is washed by inert gas. succeedingly at the b 2nd process The chlorine compound formed beforehand is made to react, the oxidizer of the gas which enables isolation of chlorine is supplied to a reactor, the chlorine which separated is discharged from a reactor, and it is characterized by washing a reactor by inert gas succeedingly.

[0017] this invention contains the reactor for enforcing the aforementioned method further.

[0018] In how to have carried out the heterogeneous catalysis of the two processes of this invention, a dee contest catalyst is used as the fixed bed.

[0019] One or more sorts of active-metal components with which it is first loaded in the form of the oxide with which a support catalyst is advantageously chosen from the group of Cu, Fe, Ru, Ce, Bi, nickel, Co, Ga, and Nd on a ceramic, the quality of silicic acid, or the support of glass, or a chloride, It consists of one or more sorts of the alkali-metal chlorides, ZnCl_2 , and/or CaCl_2 which are chosen from the group of LiCl, NaCl, and KCl. Calculating the amount of active-ingredient charge as a metaled weight rate, two to 30% of the weight, it is 5 - 20 % of the weight advantageously, and the amount of alkali-metal chloride charge is 0 - 20 % of the weight advantageously zero to 30% of the weight.

[0020] An advantageous support system uses as the base SiC, Si_3N_4 , BN, B₄C, WC, TiC, TiN, ZrN and AlN, and ceramic support especially chosen from the group of SiC and/or Si_3N_4 advantageously.

[0021] the ceramic support of a macro pore (macroporous) -- SiC [of a macro pore] and/or Si_3N_4 support is advantageous. especially advantageous.

[0022] a catalyst -- the corrosive salt melt on support (SLP (supported liquid phase) catalyst) -- for example, (CuCl₂-KCl-FeCl₃-NaCl) -- from -- it becomes, therefore a very high demand is imposed about the stability of support Reaction temperature is about 400 degrees C. To the corrosive salt melt of a catalyst, silicate and SiO₂ are not chemically stable, and produce an inadequate working life. On the other hand, the support of ceramic material fills low HCl adsorption and all demands about stability, therefore is suitable for especially the method of this invention.

[0023] Especially an advantageous catalyst consists of the Cu/Fe active-metal mixture and KCl, and/or the NaCl salt component which have SiC of a macro pore, and/or the Cu/Fe ratios 1:9-9:1 of the atom on Si_3N_4 support.

[0024] An alkali-metal chloride forms a wrap salt melt (SLP catalyst) for support together with an active metal with reaction temperature. This decreases the amount of the active ingredient discharged from a reactor, and improves an active-metal distribution. For example, especially the mixture 2 near each eutectic point, for example, CuCl, KCl, and FeCl₃ and NaCl are advantageous.

[0025] Therefore, the catalyst for which were extremely suitable consists of a FeCl₃/NaCl salt melt which has CuCl₂, KCl salt melt, and the Fe/Na ratios 3:1-1:3 which have Cu / K ratios 3:1-1:3, Cu/Fe ratios are 3:1-1:3, and all (Cu+Fe) the amounts of active-metal charge are 5 - 20 % of the weight.

[0026] A catalyst can be manufactured by the well-known method. When using a chloride precursor, i.e., a fusibility salt, manufacture by sinking in is possible. Of course, it is also possible to leave other precursors (a nitrate, a carbonate, a hydroxide, acetate, etc.). Since multiplex sinking in is generally required because of the high amount of active-metal charge, sinking [of salting-in liquid which is an easy method, namely, is different] in can attain intentionally the uneven active-metal distribution on a particle cross section. Sinking [of the first Cu salting-in liquid] in and sinking [continuing / of Fe salting-in liquid] in make it possible to manufacture a core with much Cu, and shell with much Fe in a particle.

[0027] The catalyst of the method of this invention can be manufactured as follows, for example.

[0028] In the mixed equipment advantageously equipped with the heating means, for example, a kneading machine, an extruder, or a shearing roller extruder The dispersant for inorganic [which may have a component (A) and an active ingredient] and/or a metal powder, and the epigenesis (C) Or a part for a component (C) and the epigenesis (A), and 80-250 degrees C a component (A) and (C) can be first added especially at 120-200 degrees C advantageously in the state of melting to the polymer of a component (B) together. [100-220 degrees C] 140-220 degrees C of 120-250 degrees C of 600-1800 bars of constituents mixed homogeneously (violently) can be especially fabricated by 700-1600 bars advantageously advantageous [especially advantageous especially] by compression, the knockout, or injection molding with injection molding 150-200 degrees C and the pressure of 500-2000 bars. The catalyst support fabricated by arbitrary forms or a catalyst, for example, Raschig ring, wheel mold goods and window frame mold goods -- bounding -- cam mold goods, a saddle, a star ring, and the punched mold goods of the configuration where reached and/or the rib was attached -- For example, in order to manufacture a sphere, a cube form, a cube, a cone, a pyramid, a prism, an octahedron, a pillar, a truncated pyramid, and a truncated cone, the 60-150 degrees C of the aforementioned forming cycles can be especially used at 80-140 degrees C advantageously the tool (compression molding) temperature of 40-160 degrees C.

[0029] the raw Plastic solid obtained after the forming cycle -- (a)300- 350-600-degree C 600 degrees C are advantageously processed by the pyrolysis at 400-600 degrees C especially advantageous, and 100-160 degrees C is 100-150 degrees C advantageously in the acid content atmosphere of the (b) gas, and a catalysis can remove a binder from the softening temperature of a polyacetal (component B) at low temperature especially advantageous

[0030] After removing from a mold, in order to remove a binder, a raw Plastic solid is processed in acid content atmosphere (gas). Because of the purpose of this invention, acid content atmosphere (gas) may be a gaseous pure acid at processing temperature, or you may be the mixture of an acid and carrier gas. Suitable carrier gas is air, nitrogen, or rare gas. A suitable acid is the acid which evaporates at a room temperature at a remarkable grade in an inorganic acid, for example, a hydrogen halide, a gaseous hydrogen sulfide, or gaseous processing temperature, for example, a nitric acid.

[0031] A suitable organic acid is the mixture of the acid which has less than 130-degree C the boiling point or a sublimation point by the ordinary pressure fundamentally, for example, oxalic acid, a formic acid, an acetic acid, trifluoroacetic acids, or these acids.

[0032] However, after removing from a mold, a binder is removable by processing a raw Plastic solid in a gaseous boron-trifluoride content atmosphere instead of a gaseous acid content atmosphere. Because of the purpose of this invention, a gaseous boron-trifluoride content atmosphere may be the mixture of a boron trifluoride or a pure boron trifluoride, and pure carrier gas. Suitable carrier gas is air, nitrogen, or rare gas.

[0033] Of course, it is also possible to use the adduct of the boron trifluoride which can be decomposed into a start component in reversible instead of a boron trifluoride, without a component decomposing at

processing temperature. Especially the addition compound with a boron trifluoride, the ether, for example, a wood ether, diethylether, dibutyl ether, and t-butyl methyl ether is suitable.

[0034] An oxalic acid anhydride or especially an oxalic acid dihydrate is advantageous. The glyoxalic acid is also suitable. Other suitable acids are a benzenesulfonic acid, naphthalenesulfonic acid and maleic acids, or such mixture. These can be used at a binder removal process independently together with carrier gas, for example, air, nitrogen, or rare gas.

[0035] At binder removal temperature, an acid results in a gaseous phase first, from here, acts on a binder, and is sublimated or solidified on the wall surface of the binder stripper after cooling. An acid results in a gaseous phase again at the continuing binder removal process, namely, an acid hardly leaves equipment.

[0036] It is advantageous to use it in the polar solvents which have the boiling point of less than 200 degrees C advantageously by using the aforementioned acid as a solution for acid supply. This type of especially suitable solvents are an acetone, a dioxane, ethanol, an acetic acid, and a formic acid.

[0037] Removal of the binder by acid-catalyst operation is an ordinary pressure, or can be carried out by reduced pressure (0.001-1 bar).

[0038] Generally 600-1100 degrees C of 600-1400 degrees C of Plastic solids can be succeedingly converted into the catalyst which has final intensity and a pore distribution advantageously by carrying out presintering at 600-800 degrees C advantageously especially under oxidization conditions (air), inert gas (N₂, Ar, helium), or reduction conditions (N₂/H₂, Ar/H₂). Generally a presintering process raises remarkably the porous stability and the porous degree of hardness of a Plastic solid. Generally 1-5kg (cuttinghardness) (800 degrees C) of 1.5-4kg (800 degrees C) of cutting hardness is 2-3.5kg (800 degrees C) especially advantageous advantageously. Generally, 0.05 to 5 ml/g, ml [of water absorption coefficients / 0.1-3 //] are [g / ml / 0.1-1 // g] especially advantageous, therefore they cannot spoil a degree of hardness remarkably, but can be boiled, and can cover the component of activity to a catalyst support more advantageously. It is also possible to produce the pore size distribution in the many modes (the 2 mode, the 3 mode, 4 mode, and many modes) other than the pore size distribution in the 1 strict mode by this method. This method makes it possible to manufacture the catalyst support and catalyst which have high intensity, high heat, and chemical stability. The forms where a Plastic solid is considered are all forms that can be manufactured with a granulation, rolling, compression, a knockout, or injection molding.

[0039] Generally an average aperture is determined by only the grid space between the powder particles used by the particle size of a component (A), inorganic powder, a metal powder, organic powder, and/or an active ingredient. Therefore, an average aperture and a pore size distribution are dependent on the powdered mean particle diameter and powdered particle size distribution which are used. The metal or ceramic powder of marketing by this method makes it possible to manufacture the stable porous material in the 1 mode without a crack, or the many modes, for example, a catalyst support, and a catalyst mechanically. Therefore, a narrow pore size distribution can be adjusted to a meso pore (mesoporous) or the macro pore range as occasion demands, and, generally the high pore distribution which carried out uniform distribution is produced.

[0040] in this way, the BET specific surface area of the manufactured catalyst support and a catalyst -- general -- 0.01-250m²/g -- advantageous -- 0.1-150m²/g -- especially -- advantageous -- 1-90m²/g -- it is 2-8m²/g (800 degrees C) especially

[0041] 95% of particle size has deviated from the mean particle diameter only 0 to 10% 0 to 20% 0 to 30%. especially 90% advantageously 80% the mean particle diameter of the powder of the component (A) used -- general -- 5nm - 500000nm -- advantageous -- 3nm - 100000nm -- especially -- advantageous -- 2nm - 50000nm nano crystal powder -- you may be -- a particle --

[0042] Suitable components (A) are as follows.

[0043] (I) SiC, Si₃N₄, BN, B₄C, WC, TiC, TiN, ZrN, AlN(s), or such mixture -- advantageous -- SiC and Si₃N₄ -- It SiC(s). WC, TiC, TiN, ZrN(s), or such mixture -- advantageously especially The ceramic powder and/or the (II) element which are chosen from the group of Si₃N₄ or such mixture, Beryllium, magnesium, calcium, iron, cobalt, nickel, Copper, zinc, titanium, a zirconium, vanadium, niobium, a tantalum, The metal and the silicon dioxides, or such mixture of chromium, molybdenum, a tungsten, manganese, and a cerium, Advantageously An element, magnesium, calcium, iron, zinc, titanium, the

metal of a zirconium and silicon dioxides, or such mixture -- especially -- advantageous -- an element -- The metal and the silicon dioxides, or such mixture of magnesium, calcium, iron, and zinc, They are SiC and Si₃N₄ especially. Or nature powder of silicic acid chosen from such mixture, By the case, and an element (III), copper, iron, cobalt, nickel, a ruthenium, a cerium, a bismuth, a gallium, neodymium, or such mixture -- advantageous -- copper -- Iron, cobalt, nickel, rutheniums or such mixture, the active ingredient especially chosen from the group of the chloride of copper, iron, or such mixture, or an oxide advantageously, and a case -- the (IV) element, a lithium, sodium, a potassium, zinc, or such mixture -- advantageous -- a lithium, sodium, potassiums, or such mixture -- especially -- advantageous -- the chloride of sodium, potassiums, or such mixture

[0044] when manufacturing a non-support catalyst, or a component (A) consists only of an active ingredient (III) -- or (III) -- and it consists of (IV)

[0045] Suitable components (B) are as follows.

[0046] (i) It is related only with a pyrolysis. Polymer or ethylene of polyethylene or polypropylene, The copolymer, the polystyrene copolymer, the polymethylmethacrylate copolymer, the polyethylene-oxide copolymer, the ethylene-vinyl acetate copolymers, or such mixture of a propylene, 1-butene, or an isobutene, (ii) It is related with pyrolysis and an acid-catalyst operation. A polyoxymethylene homopolymer or 50 - 100 % of the weight of copolymers well-known from the Europe (B1) patent public presentation No. 444475 specification, Advantageously whether it dissolves in 80 - 88 % of the weight, and (B-2) B1 uniformly advantageously especially 70 to 90% of the weight Or polymer of less than 1 micrometer of dispersed mean particle diameters, advantageous -- Polly 1, 3-dioxolane, Polly 1, 3-dioxane, Polly 1, and 3-dioxepane -- especially -- advantageous -- Polly 1 and 0 - 50 % of the weight of 3-dioxepane -- advantageous -- 10 - 30 % of the weight -- especially -- advantageous -- 12 - 25 % of the weight

[0047] The organic binder may consist of mixture of one or more sorts of thermoplastics, for example, a polyacetal, polyethylene, polypropylene, polystyrene, a polymethylmethacrylate and one or more sorts of plasticizers, for example, a polyethylene glycol, a polypropylene glycol, a poly butanediol formal, a phthalic ester, an ethylene-vinyl acetate copolymer, and a MONTAN ester wax.

[0048] A suitable polyacetal binder is a polyoxymethylene which has molecular weight 10000-500000 advantageously, for example. It is also possible to use a trioxane, and Polly 1 of a copolymer with ring type ether, for example, ethylene oxide, and 1, and 3-dioxolane or formal, 1 [for example,,], 3-dioxepane, 1, and 3-dioxanes or such mixture or a homopolymer, 3-dioxolane, Polly 1, 3-dioxane or Polly 1 and 3-dioxepane other than the homopolymer of formaldehyde or a trioxane, and, generally the amount of a copolymer is 10 - 30% of the weight of polymer.

[0049] Furthermore, a binder may contain an assistant, for example, a thermoplastic binder, for example, polyethylene, a polymethylmethacrylate or a polyethylene oxide and a dispersant or lubricant, for example, a polyethylene glycol, stearin acid, a fatty alcohol, a polyvinyl pyrrolidone, or polyvinyl alcohol. Generally the amounts of an assistant are 0.1 - 12% of the weight of all material.

[0050] a suitable component (C) -- a dispersant well-known from the Europe patent public presentation No. 444475 specification, for example, an organic carboxylic acid, an amine, an amide or maleimide, stearin acid, a polyvinyl pyrrolidone, polyvinyl alcohol, a polyethylene glycol, a polypropylene glycol, a polyethylene oxide, and montan wax -- advantageous -- an organic carboxylic acid, an amine, an amide or maleimide, a polyethylene glycol, and a polyethylene oxide -- they are an organic carboxylic acid, an amine, maleimide, a polyethylene glycol, and a polyethylene oxide especially advantageous

[0051] the mixture used in order to manufacture a catalyst (it mixes together) -- general -- (Component A) 15-70 % of the weight -- advantageous -- 30 - 70 % of the weight -- especially -- advantageous -- 50 - 65 % of the weight, and (Component B) 30-85 % of the weight -- advantageous -- 30 - 70 % of the weight -- especially -- advantageous -- 35 - 50 % of the weight, and a component (C) -- it consists of 2 - 8 % of the weight advantageously especially one to 12% of the weight zero to 15% of the weight

[0052] An active ingredient can be used as direct powder, in the case of a non-support catalyst, when it is a support catalyst, it can be succeedingly covered with sinking in into support material, or it can be blended together with support material.

[0053] In a manufacturing process, the start powder particle which generally carried out the solution condensation (deagglomerate) of the component (A) using the dispersant (C), and was made into the

uniform size in this way is blended with an organic binder (component B) in a comparatively high component (A) content. An organic binder fills the grid space between the powder particles arranged regularly made into the general almost uniform size. Generally the macro pore of the range of about 100 micrometers which exists in the start powder of a component (A) as a result of coagulum formation is eliminated by solution condensation. When using the powder which has narrow 1 mode particle size distribution, the pore by which it was size-sized very uniformly between powder particles remains by removal of an organic binder and an organic dispersant. When using the powder which has a multi-mode particle size distribution, or when using porosity powder, a multi-mode (2 mode, 3 mode, 4 mode, or many modes) pore distribution can be produced, and an aperture is determined by the grid space between powder particles, and the internal porosity of a powder particle.

[0054] A catalyst may be a uniform support catalyst or a non-support catalyst. A non-support catalyst consists of catalytic activity material. A support catalyst can be manufactured by covering the precursor of a catalytic activity component or a catalytic activity component with being immersed, spraying sinking in, spray drying, precipitation, a high coat (hicoating), and thinning paint (washcoating) to the catalyst support of an inactive porosity ceramic or a metal. After a catalyst sinks in advantageously, respectively, it is dried.

[0055] The catalyst acquired after pyrolysis has the 50-100000nm pore size distribution measured with the mercury pore plan by method of mercury penetration. (a) 10 - 95% of pore volume is 0.2 to 100 times the average pore diameter, 10 - 80% of (b) pore volume is 0.5 to 100 times the average pore diameter, and/or 50 - 95% of (c) pore volume is 0.2 to 1 time the average pore diameter. And/or, 50 - 80% of (d) pore volume is 0.5 to 1 time the average pore diameter, and there is less half-value width of the (e) pore size distribution than 0.5 times of an average pore diameter.

[0056] The catalyst acquired after an acid-catalyst operation and the continuing pyrolysis has the 50-100000nm pore size distribution measured with the mercury pore plan by method of mercury penetration. (a) 10 - 95% of pore volume is 0.2 of an average pore diameter - double precision, 10 - 80% of (b) pore volume is 0.5 of an average pore diameter - double precision, and/or 50 - 95% of (c) pore volume is 0.2 to 1 time the average pore diameter. And/or, 50 - 80% of (d) pore volume is 0.5 to 1 time the average pore diameter, and there is less half-value width of the (e) pore size distribution than 0.5 times of an average pore diameter.

[0057] Support has the raised degree of hardness. This high degree of hardness enables multiplex sinking [of the catalytic activity component to a catalyst] in, therefore enables charge of a high active metal. The catalyst manufactured has the very good water absorption coefficient of the range of 0.1 - 1.0 ml/g, and can cover the component of activity more compared with the conventional support.

[0058] If a chloride is used in the case of an active ingredient, manufacture by sinking in is possible. It is also possible to, leave other precursors (a nitrate, a carbonate, a hydroxide, acetate, etc.), of course.

[0059] Since multiplex sinking in is generally required for charge of a high active metal, salting-in liquid which is an easy method, namely, is different in the uneven active-metal distribution on a particle cross section can be intentionally attained by sinking in. It makes it possible to obtain a core with much Cu, and shell with much Fe in a particle by infiltrating Cu salting-in liquid first and infiltrating Fe salting-in liquid succeedingly.

[0060] The fixed bed other than a catalyst can also contain the layer of back or the front. The layer of the back which consists of support material which doped a pure support material, the support material which doped the alkali-metal chloride, or Cr is advantageous.

[0061] furthermore, the catalyst bed itself may consist of stage attachment ***** of a different catalyst arranged continuously, and it can also be changed to each fraction in the amount of active-metal charge, and (simultaneously) an active-metal distribution

[0062] Down stream processing of this invention is expressed as an example of the following Cu catalysts simplified extremely.

[0063] The hydrogen chloride introduced into the fixed bed at the 1st process of chlorination is chemically combined with a catalyst, this exothermic reaction produces isolation of water, and the water which separated is discharged from a reactor.

[0064] $\text{CuO} + 2\text{HCl} \rightarrow$ Separating again the chlorine accumulated at the 2nd process of $\text{CuCl}_2 + \text{H}_2\text{O}$ oxidization oxygen content gas and by adding oxygen or air advantageously, this endothermic reaction

forms the chlorine gas stream which hardly contains water and a hydrogen chloride.

[0065]

$\text{CuCl}_2 + 0.5\text{O}_2 \rightarrow \text{CuO} + \text{Cl}_2$ -- the reaction shown here explains the principle of a continuous reaction -- it is not alike too much. This reaction principle is applicable to other suitable decontamination catalysts. It is known by the intermediate product in which the reaction process actually produced to all catalyst systems is formed that it is quite complicated substantially.

[0066] The method of this invention can be advantageously enforced using one fixed bed reactor or two or more fixed bed reactors which were connected. The number of reactors and arrangement are mainly determined by reaction dynamics, and are determined by demand of the catalyst and the method that this is used further. For example, total yield can be optimized by increasing the number or size of a reactor in the limited partial process.

[0067] A drawing explains the method of this invention. Five connected fixed bed reactors are shown in drawing 1 as an example. About 300-450 degrees C preheated all the fixed bed reactors at 340-420 degrees C advantageously at the time of the start of the method of this invention.

[0068] The following processes advance continuously in each fixed bed reactor.

[0069] 1. It is ambient temperature, in a request, condense a chlorination hydrogen chloride at the raised temperature, or, in a request, dilute with inert gas, and introduce into the fixed bed reactor which now is in chlorination mode through a supply line 1. the 1st portion of the fixed bed, and the outlet portion of a reactor -- a catalyst bed -- or you may consist of a floor of an inactive material advantageously (about 20-100cm) HCl used in a reactor entry-zone region -- above the floor level [hot] -- accumulation -- reaction temperature -- 300-450 degrees C is especially heated at 340-420 degrees C advantageously. The HCl gas which it preheated reacts in the catalyst bed of a metallic-oxide component and back, forms a corresponding chloride, and separates the reaction water to produce. A HCl reaction moves a catalyst bed in the form of an advantageous sharp reaction front about process engineering. The generated heat of reaction is accumulated at a catalyst bed. Thereby, a temperature front moves a reactor behind a reaction front. A local temperature rise is determined by the local concentration of the active ingredient which mainly reacts in a catalyst. In the method of this invention, the range of about 10-90 degrees C of temperature rises is about 30-60 degrees C advantageously. Composition of a catalyst and the inactive content of a floor can adjust this. The first chlorination temperature is doubled with the chlorine accumulation capacity of a catalyst so that it may not exceed advantageously about 380-450 degrees C of maximum temperatures of the low range from 410 degrees C in the hottest portion of a fixed bed reactor toward the end of chlorination. This prevents risk of happening of evaporation of a changes chloride. The heat generated in a reaction is accumulated at a reactor, and remains. Only the temperature gradient of almost the above [the temperature of a catalyst bed] rises in a reaction. The temperature characteristic which rises toward a reactor outlet is adjusted in a reactor. The fixed bed functions as a reproduction thermal accumulator. Heat of reaction is accumulated as a result of the aforementioned temperature rise under chlorination at the fixed bed. This heat accumulated at the fixed bed supplies heat of reaction required for dechlorination by the continuing endothermic. Since the heat of reaction generated during chlorination is larger than heat of reaction required for dechlorination, the heat dissipation discharged in heat loss and a washing process can be offset. Generating of heat and exhaustion of heat are mutually in agreement advantageously with the temperature rise by this invention so that external heating or external cooling may not be required. The amount of the chlorine accumulated at the fixed bed mainly reaches with the active-ingredient content in a catalyst, and is determined by the amount of the catalyst in a reactor. It can be increased simply and at a low price by the amount of the chlorine accumulated for every reactor for easy structure by what (it lengthens) a reactor is expanded for. Although the duration of chlorination is not indispensable, each circulation can prescribe preferably the charge time on which it decided [in / industrial operation / it is the same and], and before this loads with a catalyst bed completely by chlorine, it is chosen so that supply of HCl may be completed. The end of a chlorination process can be easily inspected by measuring HCl outlet concentration. The steams which remain in reaction water or a reactor are collected by the line 2, and are discharged. 300-450 degrees C of start temperature of the catalyst bed of chlorination should be 340-420 degrees C advantageously.

[0070] It is desirable that HCl which it does not chlorinate completely to exsorption (breakthrough) of

HCl, it is interrupted at the suitable early time, and still more sufficient unreacted metallic oxide remains to a reaction outlet field, and passes in addition and blows a catalyst bed especially while a washing process is going on joins together by the reaction.

[0071] Alternatively, two or more reactors connected continuously make it possible to pass over exsorption of HCl and to continue HCl charge, supply the HCl content gas which leaves the 1st reactor to the 2nd reactor after HCl exsorption, and react completely here. The 1st reactor can be used for a washing process after the end of the charge which the 1st reactor followed. Washing gas is supplied to one reactor in chlorination mode. HCl supply is changed to the 2nd reactor and the remains HCl which leave the 2nd reactor are supplied to the 3rd reactor. Therefore, at a industrial process, in this deformation, at least three fixed bed reactors are used, there are at least two reactors connected continuously in chlorination mode, and one reactor is in dechlorination mode. In the arrangement which uses only three reactors, each reactor performs a washing process following a reaction process in time. However, in order to attain the gas stream stabilized more, it is more advantageous to use more numerical-response machines. You have to adjust each process in time mutually so that a reactor may be periodically changed to each continuing mode repeatedly (an interval is opened in time or it is advantageously). By continuation of the HCl charge which passed over HCl exsorption, the chlorine incorporation of a catalyst increases, therefore the availability of a catalyst increases. Discharge of the chlorine which continues from the catalyst which loaded with many chlorine advances more quickly, and produces the higher chlorine content in a dechlorination gas stream.

[0072] 2. In addition, unreacted HCl exists in the gaseous phase of a reactor after a washing process charge end. You have to remove this before the continuing dechlorination. For this reason, a reactor is washed by inactive washing gas, N₂ [for example,], through the washing gas line 3. Chlorination is completed before HCl exsorption. In addition, it remains, and the band which has sufficient chlorine accumulation capacity for the outlet field of a reactor by this reacts with the remaining HCl washed by washing gas through the reactor, and accumulates the chlorine of a corresponding amount. This operating instruction prevents that HCl is carried by the exterior of a reactor in a washing process. The time of a washing process is several times the residence time, if HCl and water stop almost existing in a reactor outlet, it will end, and it is decided by measurement. Although washing time is not indispensable, in the long-term operation of equipment, it is always preferably the same. After inert gas leaves a reactor similarly, it is taken out through the collection line 2. washing -- a parallel flow (cocurrent) -- or although it can carry out by the counterflow, it is advantageous to carry out at a parallel flow, i.e., the same flow direction as chlorination

[0073] By changing the amount, washing time, or temperature of washing gas, a washing process can be used, and superfluous heat of reaction can be removed from a reactor, or additional heat can be introduced into a reactor.

[0074] After each reaction process, a reactor is advantageously washed by the parallel flow or the counterflow for 0.2 - 5 minutes for 0.1 - 20 minutes using suitable inert gas, N₂, CO₂, and CH₄, or rare gas. [for example,]

[0075] 3. A dechlorination reactor shows the temperature characteristic which rises toward a reactor outlet as a result of exoergic chlorination. Dechlorination is endothermic nature and is carried out at desirable high temperature. For this reason, the flow which passes a fixed bed reactor in a dechlorination process exists in an opposite direction advantageously. About 300-450 degrees C of 360-420 degrees C of temperature under dechlorination should be 380-410 degrees C especially advantageous

advantageously. For this reason, an oxidizer (air, oxygen, or other oxidization gas, for example, N₂O) is introduced into another edge of a reactor through the oxidization gas line 4, and it heats to reaction temperature all over an inactive floor here. Isolation of chlorine moves a reactor in the form of a reaction front similarly with oxidization and it of a catalyst. Dechlorination is endothermic nature. Energy required for a reaction is supplied from the heat content of the fixed bed. Thereby, cooling of an about 10-90-degree C catalyst bed arises. Catalyst composition and the inactive content of a floor can adjust a temperature change. The catalyst bed has regained a great portion of temperature characteristic before chlorination after dechlorination. The end of chlorine isolation can be supervised by measuring the chlorine content of a product gas stream. The level of chlorine in the amount of the chlorine from which per unit time was isolated, therefore the chlorine gas stream of a reactor outlet decreases with time.

Shortly after the level of chlorine falls below to the minimum value, it is desirable to interrupt isolation of chlorine. Yield can be raised at the time of empty - which can be attained by this. In a continuous method, the time of chlorine isolation is maintained uniformly or it adjusts through measurement of a chlorine content. A chlorine gas stream is cooled, it collects by the product gas line 5, and a tail end process is supplied.

[0076] 4. It is filled up with the gas space in the fixed bed of a reactor after washing process dechlorination by the oxidizer and the chlorine which separated. Before resuming chlorination, it is required to wash a reactor by the inert gas again dried through the line 3. In order to exchange a several times as many gaseous phase as this, washing time is several times the residence time. It is the same as dechlorination, or washing can be carried out in the opposite flow direction. If reactor outlet gas does not contain chlorine any longer or the level of chlorine falls below to the aforementioned threshold value, the end of a washing process will be reached.

[0077] In order to put equipment into operation, the air or inert gas which preheated is supplied to the fixed bed through a line 6 and a heat exchanger 7, and the fixed bed is heated to reaction temperature.

[0078] About 0.5-20 bars of pressures of the method of this invention are about 1-10 bars advantageously, and the pressures of each process may differ.

[0079] Chlorination and dechlorination are carried out continuously periodically, and as the heat accumulated in a chlorination process at a catalyst supplies reaction energy required for endothermic nature dechlorination, it can dechlorinate following chlorination.

[0080] Having even about 420 degrees C of remarkable vapor pressure has from reference the well-known metal chloride formed by the method of this invention. Drawing 2 shows the temperature dependence of the vapor pressure of CuCl_2 as an example. Numeric value It is indicated by Allen J.A. Clark A.J. Rev. Pure and Appl. Chem. 21 (1971) 145-166 page. For this reason, it is because otherwise, and the catalyst of it which can avoid temperature quite higher than 420 degrees C during dechlorination deactivate. Generally it is desirable to dechlorinate at 370-420 degrees C advantageously about 300-450 degrees C depending on catalyst composition.

[0081] The temperature under chlorination should be chosen as a function of catalyst composition, and after exothermic reaction is completed, it is made not to exceed the temperature of about 380-430 degrees C which is the highest allowable temperature for the volatility of a metallic oxide.

[0082] In consideration of the special demand of a method medium very corrosive in the reactor of this invention which enforces the method of this invention, some of media exist as a molten-metal chloride under method conditions.

[0083] In the prolonged examination, the unsuitable thing made the reactor of ceramic material clear for the demand by which even a high-grade alloy like Hastelloy (Hastelloy) C is not suitable for only a molten-metal chloride and short time contacting, and is imposed on the fundamental elasticity of a reactor.

[0084] It became clear that the metal reactor which the interior lined with an acid-proof ceramic material fits this method extremely. Although the case where the whole interior which reacts is lined is the safest, it is desirable to line only the band containing a catalyst or an inactive floor from the reason of costs, and it is because the volatile metal chloride melt which is easy to react on it, that is, a metal to an early attack arises only in this quite hot band.

[0085] A suitable ceramic material has composition of $2\text{O}324$ - 26% of commercial acid-proof aluminosilicates, for example, aluminum, $2\text{O}70$ - 74% of Si, 21 - 2% of $\text{TiO}(\text{s})$, and $2\text{O}31$ - 2% of $\text{Fe}(\text{s})$. In order to manufacture lining for the melting brick manufactured from this material, inorganic acid resistance is combinable with a commercial binder, for example, a potassium water glass binder, similarly.

[0086] Generally the thickness of lining is 5-10cm advantageously about 1-30cm.

[0087] The lining brick did not penetrate gas completely but it became clear that a metal chloride melt is spread to some extent in a lining brick. However, the depth of penetration is small. When using a commercial acid-proof lining brick, a melt permeates only the surface layer of a lining brick. Even when stress cracking is formed, a metal chloride melt cannot be given to a reaction wall. In lining, the temperature gradient to which temperature falls toward a reaction wall exists. The metal chloride melt which permeates lining, and is cooled and solidified is not corrosive any longer in the state where it

solidified, therefore produces synthesis of a crack simultaneously.

[0088] The entrance and outlet of a reactor do not exist with reaction temperature. Reproduction formula accumulation mainly performs heating and cooling of reactant gas all over the inactive floor arranged the front and behind a catalyst bed, respectively. The inversion of a periodic flow guarantees that the heat introduced first remains in a fixed bed reactor, and is hardly discharged by the gas stream in a starting process. A molten-metal chloride does not exist in the exterior of a catalyst bed substantially as a result. It became clear that a special alloy like Hastelloy C is strong to change of composition of a gaseous phase. That all manufacture the reactor Plastic solid of a special alloy requires cost, and it is unnecessary. It made the advantageous manufacture method clear that it is use of the ordinary steel coating for boilers which used special steel. The shell of the outside of a cylinder reactor (for example, a length of 8m, the diameter of 2m) is used for guaranteeing required intensity, and may consist of ordinary steel for low-alloy boilers like H1 or H2. For example, it uses for covering inside the reactor using the special alloy C with a thickness of 2-5mm, for example, a Hastelloy, Inconel (Inconel) 600, or the corresponding alloy guaranteeing the corrosion resistance to a gaseous-phase component. Lining for a cylinder core of a reactor guarantees the corrosion resistance to a metal chloride melt. A suitable acid-proof melting brick is chamotte brick (aluminosilicate) combined, for example by the suitable acid-proof sexual conjugation agent (for example, potassium water glass). Lining equipment is further improvable by blending a ceramic-fiber mat advantageously.

[0089] The reactor may have the size of arbitrary requests in principle. Advantageously, in order to guarantee stability and to avoid the unnecessary thickness of a reaction wall, a diameter is chosen so that it may not become quite larger than 2m. In order to attain a sharp reaction front, the ratio to the diameter of length is large and should be advantageously chosen from 2:1 greatly from 4:1. The length of a reactor is limited by the fall of a pressure. For operating continuously, you should connect at least two reactors in parallel. However, in order to enable exchange of a reactor, without equalizing continuous manufacture, and under use giving big flexibility, offering the free space of each reaction process (chlorination, a washing process, dechlorination), and barring manufacture, it is advantageous to use three or more reactors which are connected in parallel and changed periodically. The transition metals which exist advantageously by it, that is, the oxidation state which are carried out with the air which preheated are not volatility, therefore heating of a reactor is because quite high temperature (higher than 400 degrees C) is borne. However, the temperature chosen must be so low that the chemical reaction of support material and transition metals is not produced.

[0090] the method of this invention -- technical --** in easy -- it can make it possible to attain nearly perfect HCl inversion by the economical method, and a complicated tail end process can be avoided As a result of the inversion of the periodic flow of a gas stream, the fixed catalyst bed functions as a heat regenerator. This excludes removal of advantageously complicated additional heat tracing or heat, and decreases total-energy consumption and the cost of a reactor remarkably.

[0091] The reactor of this invention is easy and cheap, it can manufacture, and high operating time is offered in the method of this invention.

[0092]

[Example]

0.590kg (20 % of the weight of $\text{FeCl}_3 + \text{KCl(s)}$ on support) of example 1 catalysts was insulated in the product made from a quartz, and it has arranged to the heated experiment fixed bed reactor. All weight %s are related with all material. It is HCl at about 360 degrees C to a catalyst succeedingly. It loaded with 11.7 l/h (reference condition) (chlorination), washed to HCl non-** succeedingly using N_2 , and oxidized for 20 minutes after that using pure oxygen 12 l/h (reference condition) (dechlorination). The reactor was again washed by N_2 before the following chlorination, and O_2 and Cl_2 were removed.

[0093] ΔT expresses the temperature change of the center of the fixed bed covering the indicated reaction time.

[0094]

[Table 1]

	時間 [s]	塩素 放出/ 取り込み [Cl モル]	空/時収率 [塩素 g/h / 触媒 kg]	ΔT [°C]
1. 塩素化 *1	1195	1.67	303	80
1. 酸化	1200	0.24	42	-16
2. 塩素化	234	0.33	303	56
2. 酸化	1200	0.21	38	-14
3. 塩素化	203	0.28	303	52
3. 酸化	1200	0.22	39	-6
4. 塩素化	226	0.32	303	54
4. 酸化 *2	1200	0.22	40	-23
5. 塩素化	187	0.26	303	48
5. 酸化 *2	1200	0.24	44	-11
6. 塩素化	181	0.25	302	45
6. 酸化 *2	1200	0.25	44	-14

[0099] *1 Chlorination *2 of the beginning after oxidizing a catalyst all night In the example of oxidization 4 fixed-bed tubular type reactor which used air 60 l/h (reference condition), about 20ml (6.5 % of the weight of Cu(s) of a SiC granular lifter, 8.1 % of the weight of Fe(s)) of catalysts for which were especially suitable was used. At 380 degrees C, the catalyst was loaded until HCl exsorption produced HCl of a pure gas in space-velocity 750h-1. Chlorination time is 6.5 minutes and washed the reactor for 2.4 minutes by N2 succeedingly. Dechlorination (catalyst oxidization) was carried out at 400 degrees C space-velocity 600h-1 using pure oxygen. Dechlorination time was 6.5 minutes. Product gas contained more Cl2 maximums than 27 capacity % and Cl2 average 23 capacity %. The reactor was succeedingly washed for 4 minutes by N2. It calculated by perfect circulation and yield, slightly high temperature, and the short cycle time were attained by the improved catalyst at the time of empty - higher than chlorine 100 g/h / catalyst kg.

[0100] Chlorination-oxidization circulation was repeated 32 times using the amount of charge, reaction temperature, and charge speed of a different grade. The result which always corresponds was obtained. The reduction in catalytic activity was not found out.

[0101] Carried out operation of example 5 experiment like Example 4 using the corresponding catalyst, however charge temperature was 425 degrees C. The dechlorination which used O2 (5 l/h reference condition) was continued until the chlorine content in reactor outlet gas fell below to 10 capacity %.

[0102] (a) It loaded until HCl exsorption arose 25% of concentration in N2 using HCl (after 6 minutes). The chlorine content in a product gas stream fell below to chlorine threshold value 10 capacity % after [of dechlorination time] 5 minutes. The maximum chlorine content was 20 capacity %, and the average chlorine content over dechlorination time was about 12 capacity %.

[0103] (b) It passed over HCl exsorption 50% of concentration in N2 using HCl, and charge was carried out for 40 minutes. The level of chlorine in a product gas stream fell below to chlorine threshold value 10 capacity % only after [of dechlorination time] 7.8 minutes. The maximum chlorine content was 50 capacity %, and the average chlorine content over dechlorination time was about 33 capacity %.

[0104] Therefore, if charge time is long, a catalyst will absorb much chlorine; the level of chlorine of a dechlorination process increases by this, and a catalyst availability increases.

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TITLE: Producing chlorine@ by separate chlorination and
dechlorination stages - by introducing gas, water and
chlorine cpds. to react with Deacon catalyst, discharging
water, introducing gaseous oxidant to liberate chlorine@,
discharging and purging reactor with inert gas

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PATENT-ASSIGNEE: BASF AG[BADI]

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BASIC-ABSTRACT:

The prodn. of Cl₂ from gas contg. HCl in a reactor contg. a Deacon catalyst (I) at elevated temp., pref. 300-450 deg. C, is carried out in 2 stages. In a 1st stage, the gas is introduced, water and chlorine cpds. fixed to (I) are formed by reaction between (I) and HCl, when the temp. rises by 10-90 deg. C, the water is discharged and the reactor is purged with an inert gas at the end of the stage. In a 2nd stage, a gaseous oxidant is introduced, which liberates Cl₂ from the Cl cpds., the Cl₂ is discharged and the reactor is purged with an inert gas.

Also claimed is the use of reactors, in which the zone contg. the catalyst bed is lined with acid-resistant ceramic materials, for Cl₂ prodn. by this process.

ADVANTAGE - The process is simple to operate, and uses a stationary catalyst bed with the aggressive salt melt as supported liquid phase, which avoids drawbacks associated with fluidised beds.

CHOSEN-DRAWING: Dwg.0/2

TITLE-TERMS: PRODUCE CHLORINE@ SEPARATE CHLORINATED
DECHLORINATED STAGE
INTRODUCING GAS WATER CHLORINE COMPOUND REACT
DEACON CATALYST
DISCHARGE WATER INTRODUCING GAS OXIDANT LIBERATING
CHLORINE@
DISCHARGE PURGE REACTOR INERT GAS

DERWENT-CLASS: E36 J04

CPI-CODES: E31-B02; J04-E01; N02; N03;

CHEMICAL-CODES:

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Fragmentation Code

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A430 A544 A758 A760 A940 C017 C100 C108 C550 C730
C810 M411 M730 M903 Q421

Chemical Indexing M3 *02*

Fragmentation Code

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